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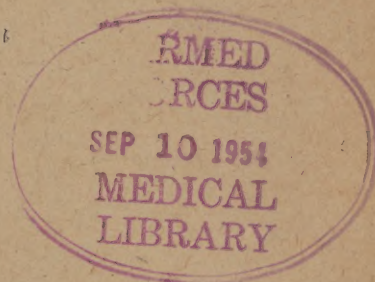
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**MANUFACTURE OF INSECTICIDES, INSECT
REPELLENTS, RODENTICIDES
I.G. FARBENINDUSTRIE A.G., LEVERKUSEN
AND ELBERFELD**

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**COMBINED INTELLIGENCE OBJECTIVES
SUB-COMMITTEE**

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MANUFACTURE OF INSECTICIDES, INSECT REPELLENTS
RODENTICIDES

I.G. FARBENINDUSTRIE, A.G.
LEVERKUSEN AND ELBERFELD, GERMANY.

22 - 24 April, 1945

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COMBINED INTELLIGENCE OBJECTIVES SUB-COMMITTEE
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MANUFACTURE OF INSECTICIDES, INSECT REPELLENTS, RODENTICIDES
I.G. FARBENINDUSTRIE, A.G.
LEVERKUSEN AND ELBERFELD, GERMANY.

1. LAUSETO OLD

Lauseto old is an analog of DDT.

a. Process of Manufacture

(1) Active Principle

To 1550 kg chloral is added 1720 kg chlorobenzene and 625 kg benzene. Then at 25-30°C in 4 hours is added 4200 kg 96% H_2SO_4 , and after 8 hours stirring at the same temperature, 1300-1500 kg 65% oleum. After 12 hours stirring the batch is poured onto ice, the acid aqueous solution separated from the crude Lauseto, then freed from excess chlorobenzene by blowing with steam, neutralized with soda and freed from the aqueous layer by settling.

Yield: 2150-2300 kg Lauseto - active principle.

Production - 120 tons/mo.

(2) Special Emulsifier

1200 kg emulsifier BX is dissolved in
2400 kg water, then is added
1800 kg emulsifier W conc and
146 kg 78% sulfuric acid

and after good stirring allowed to stand 6 hours. The aqueous solution is withdrawn and neutralized exactly with 25% ammonia. After additional standing, the aqueous liquid is withdrawn.

Yield: 3200 kg

BX is butylated naphthalene sodium sulfonated

W Conc is a mixture of 60 pts oxyethylated fore-runnings from the ethylphenoldodecylene manufacture with 30 pts aqueous paste of phenyl pentaglycol ether sodium sulfonate.

(3) Addition to Lauseto

55 pts Lauseto - active principle - is dissolved in
24 pts trichlorethylene, and at 40°C
6 pts paraffin oil and
16-18 pts special emulsifier is added, stirred a few
hours and allowed to stand.

After separating a small amount of aqueous liquid the
Lauseto is ready.

Omega Powder

4% Lauseto - active principle
34% Chalk
62% Talcum

(b) Method of use of Lauseto Old

Material for protection of garments against the occurrence
of clothes lice.

Use: For impregnation of a set of clothes consisting of 1 shirt,
1 set of underclothes, 1 pair socks and 1 sweater (about 1.5 kg)
three tablespoons of Lauseto is necessary.

Practical Directions: Three tablespoons full of Lauseto are put
into an empty pail and 3 liters cold water poured in slowly with
stirring. A white emulsion is formed in which are placed the
pieces of clothing after washing and wringing, which are allowed
to remain therein for 15 minutes with kneading. They are then
wrung out and dried in the air (not in an oven).

This treatment gives protection against lice for 3 months
so long as the clothing is not boiled. Washing with water warm
to the hand does not affect them. After three months and after
every boiling, they must be reimpregnated.

The liquid pressed out of the clothing is caught in a
pail and used further. For each set of clothes a mixture of
2 tablespoons of Lauseto and 1.5 liters of water are added, but
after the fourth addition the liquid is thrown away.

For the treatment of greater amounts of clothing at one
time, the following consumption is calculated:

100 sets need about 3 liters Lauseto and 300 liters water
further 100 sets " " 2 " " " 150 " "

To protect uniforms, coats, woolen coverings, etc., additional spraying of 1-2% Lauseto emulsion by means of Flit sprays is recommended besides the impregnation of the body clothing.

In winter with long storage in the cold, Lauseto becomes thickish. Neither this nor a small separation of crystals has any effect on the action. Lauseto packages should be placed at room temperature for 24 hours before use, or if metallic, put in warm water to make ready for use.

Drs. Rüsck, Stotter and Wenk were interviewed.

2. OTHER DDT TYPES.

a. Gix

Gix is made at I.G. Hoechst and is reported here for the sake of completeness. Aniline is diazotized in the presence of copper and hydrofluoric acid to give the phenyl diazonium fluoride which splits out nitrogen to give fluorobenzene. From this point the process is similar to DDT using fluorobenzene in place of chlorobenzene. Gix is 7 times better than Gesarol (DDT) on flies but costs 10 times as much.

Dr. Bayer was interviewed.

b. Me 1700

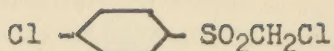
Me 1700 is made from chlorobenzene and dichloroacetaldehyde instead of chloral. Its properties are practically equal to Gesarol but it is more difficult to make. It cannot be produced without some trichlor compound and therefore comes under the Geigy patent.

Dr. Meiser was interviewed.

c. General

No other substitution compounds approach the effectiveness of Gesarol or Me 1700. Nitro, acetyl, sulfonamid, etc., substitution of chlorine in the rings or of other halogens on the other end of the molecule and lengthening of the chain all result in less active compounds except for Gix above.

3. LAUSETO NEW



Mol. Wt. 225 FP 122°C

a. Manufacture

The following is a laboratory method of manufacture:
840 gms chlorobenzol is dropped into 3.6 kg chlorosulfonic acid at room temperature and heated 1 hr to 80-90°C. After cooling the mass is poured on ice and the sulfonechloride filtered and washed. Yield 80% of theory.

1 kg sodium sulfite 90% in 4 liters warm water is added in small portions at 70°C to the raw sulfone chloride and 1.2 kg 45% NaOH run in to pH 9. After 2-3 hrs at 80°C impurities are filtered off and the sodium p-chlorosulfinate separates in 85% purity. The sulfinate in solution can be obtained either through salting out or acidification. Yield about 70% of theory.

At Leverkusen because they had no sodium dichloracetate, the latter was prepared from chloral with Na_2CO_3 and a trace of NaCN. To the alkaline solution of sodium dichloracetate from 294 gm chloral (90% yield), 420 gm p-chlorobenzene sodium sulfinate is added and stirred at 95°C. After an hour the condensation product begins to separate and after standing overnight is filtered and washed alkali free. Yield 80-89% on sulfinic acid.

The same general procedure was followed in the plant, except that zinc dust was used for the reduction instead of sodium sulfite. The chloral is heated in a kettle with soda ash and 1% sodium cyanide. The chlorsulfinate is added in the same kettle. The batch is heated to 75°C, the compound is precipitated and filtered. Production capacity is 20-30 tons active material.

b. Use

Lauseto new is five times as active as Gesarol against lice. It is also active against bed bugs but of little value against flies and aphids. It is prepared in two forms.



(1) An aqueous suspension of active material, bentonite and the emulsifying agent used in Lauseto old.

(2) Tablets weighing 25 gm containing 44% active material plus bentonite, talc and chalk.

The suspension is used for impregnating clothes at the rate of 10 gm/liter. The tablets are pounded, then suspended in 5 liters water. One is sufficient for one soldier. Lauseto new is removed from clothes by washing but is less affected by heat than Lauseto old, particularly by ironing.

Drs. Redies, Bayer, Wenk, Rüsich, Bonrath, Stotter were interviewed.

4. D 1210

D 1210 is a mixture of ω -chloromethyl phenylsulfone and 3,4 dichlorobenzyl alcohol, i.e.  $\text{SO}_2\text{CH}_2\text{Cl}$ and Cl  $\text{SO}_2\text{CH}_2\text{OH}$

The chloromethyl phenyl sulfone is made in an analogous manner to Lauseto new starting with benzene instead of chlorobenzene.

For the 3,4 dichlorobenzyl alcohol 130 kg paraformaldehyde, 2500 kg pure o-dichlorobenzene and 1250 kg pure zinc chloride are heated to 60°C and dry HCl gas passed in. The batch is washed 3-4 times with water, the last wash being weakly alkaline. The water is drained and the product dried. Yield 75% of theory.

60 kg of 3,4 dichlorobenzyl chloride of BP $104-127^\circ\text{C}$ at 6 mm is heated to $95-100^\circ\text{C}$ with 250 kg 10% soda ash solution with rapid stirring for 40 hrs to get 95% of the side chain chlorine split off. After cooling, the oil is separated from the water and heated to 110°C . At this temperature 1 kg ammonia gas is passed in to convert residual chloride to amine which is washed out with dil. H_2SO_4 . The oil is washed with water and dried at 100°C . Yield 85% of theory.

1 pt (vol) D 1210 is stirred well with 0.5 pts (vol) water warmed to 70°C , and the clear solution stirred into an emulsion with 8.5 pts spring water.

D 1210 is the best preparation against bed bugs, has some effect on lice, but is not effective on flies or mosquitoes. It can be sprayed in rooms or objects can be treated by dipping or washing. Windows should be kept open until the emulsion dries and objects should be dried in the open air.

Drs. Rüsich, Wenk, Bonrath and Stotter were interviewed.

5. BLADAN

Bladan is the hexa ethyl ester of tetra phosphoric acid $((C_2H_5O)_2PO)_3PO$. For manufacture 1 mol phosphorus oxychloride is mixed with a little more than 3 mols triethyl phosphate and blown to the top of a column packed with Raschig rings with a reaction kettle at the bottom, the system being kept at 30 atmospheres pressure and $140^{\circ}C$. The product flows to a cooler of lead coil.

For use, a mixture is made of 60% Bladan, 20% wetting agent (Nekal is not satisfactory) and 20% solvent such as xylol. Bladan is particularly applicable to plant lice and is more active than nicotine. However, it does slowly decompose in dilute solution though not too quickly to be ineffective if not stored for a long time.

Drs. Bayer, Klebert, R"usch, Wenk, Bonrath and Stotter were interviewed.

6. LUCEX (PENTAÖL)

Lucex is made by chlorinating the side chain of ethyl chlorobenzene.

(a) Manufacture

560 kg dry pure chlorobenzene and 38 kg fine aluminum chloride are treated over 20 hrs with 70 kg ethylene gas in an iron or lead lined kettle with a stirrer at a temperature of $90-95^{\circ}C$. The ethylene can be pure or a 50% gas. After stirring for a few hours more, the batch is treated with 2000 liters water and cooled to $50^{\circ}C$. A small amount of HCl in the water prevents emulsion.

The crude chloroethylbenzene is fractionated as follows:

Forerun $130^{\circ}C$. (chlorobenzene)	50%
Intermediate $130-180^{\circ}C$	7 - 10%
Ethylchlorobenzene $180-204^{\circ}C$	30%
Diethylchlorobenzene over $204^{\circ}C$.	7%
Residue	3%

The ethylchlorobenzene is heated to 140°C in a 250 liter porcelain or pure nickel apparatus and chlorinated under the light of a mercury lamp to Sp. Gr. 1.600-1.605/20°C. As chlorination becomes slow, temperature is raised to 160°C and the batch finally blown with air.

Yield 200-300% by weight of the starting material.

Analysis 66.25% total chlorine

b. Use

Lucex powder is prepared as follows:

4.0% PentaBl
0.3% Chlorsulfone
34.0% Chalk
61.7% Talc

Lucex is not as effective as Lauseto new but is cheaper. It does have some odor.

Drs. Bayer, Redies were interviewed

7. MOSQUITO REPELLENT 50/181

Trichloracetyl chlorethylamide, $\text{CCl}_3\text{CONHC}_2\text{H}_4\text{Cl}$ was made only in the laboratory during February and March, 1945. Chlorethylamine is prepared by treating ethanolamine with thionyl chloride. The product is reacted in aqueous solution with the methyl ester of trichloroacetic acid. After short boiling, the chlorethylamide separates in practically pure form.

The repellent solution has the following composition:

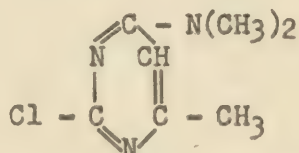
7.50% Trichloracetyl chlorethylamide
1.25% CaCl_2
1.25% MgCl_2
60.00% Absolute alcohol
30.00% Water

It has been tested against one strain of culicine mosquitoes but not against anophelines or other biting insects. No skin irritation has developed on those who have used it so far. When applied to skin, clothes and socks, the repellent effect lasts for about 6 hours. It is definitely superior in action to diethyl phthalate. Other phthalates do not seem to have been tried.

Drs. Rüsck, Bonrath, Stotter and Bayer were interviewed.

8. RODENTICIDES - ELBERFELD

a. "Castrix giftkörner" is a rodent poison to be used in place of thallium compounds. It is



and is made from acetoacetic ester, urea, followed by chlorination and amination. It is very toxic to mice, but low in toxicity for domestic animals.

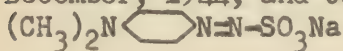
1 part of the active principle is put with 1000 pts wheat grain and production capacity for the mixture is 30 tons/mo at Elberfeld.

"Zelio-Körner" contains 2% thallium sulfate per 1000 pts wheat grain. Capacity is same as for Castrix but not simultaneous.

Drs. Lutter, Rietz were interviewed.

b. Gp 4243

In order to replace thallium compounds as rat poisons a new rodenticide Gp 4243 of which the active principle is p-dimethylamino phenyl diazo sodium sulfonate was developed in December, 1944, and January, 1945. The formula is



Process

53 gm dimethylanilin (99.5%) is treated under 5°C with 150 cc hydrochloric acid and 100 cc 30% sodium nitrite in a total volume of 500-600 cc. After an hour's stirring the batch is filtered and washed with dilute HCl. The nitroso derivation is reduced gradually with 300 gm iron powder and 60 cc of 50% acetic acid with heating, made alkaline and filtered while still hot, giving 1200 cc weakly lilac-colored solution. 200 cc of this is made acid to Congo red and decomposed with 10 cc HCl in presence of ice and diazotized at 0°C with 4.6 gm sodium nitrite. 30 cc of 20% soda solution is added with 10 gm anhydrous sodium sulfite and then a further 35 cc soda solution. The diazo sulfonate is precipitated with 50 gms salt, filtered and dried. Yield 22 gm or 132 gm for the whole batch.

Use

The formula for use is:

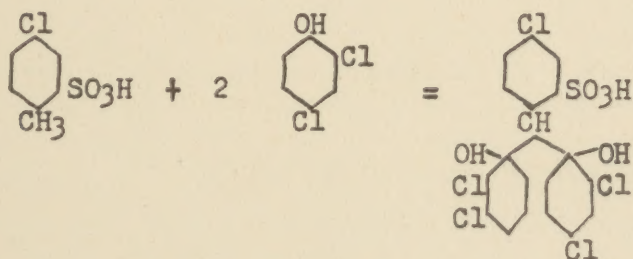
6.0% active material
0.2% Permanent Red R Extra
0.4% Fast red (Echtrot) A.V.
93.4% Bran

Tests were being carried out to determine if 3% active material was satisfactory in the formula.

9. EULANS (MOTH REPELLENTS)

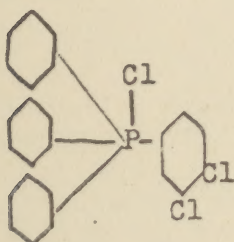
The Eulans are old having been introduced in 1927. Some of the newer varieties are:

Eulan New

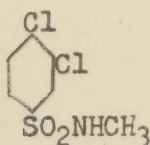


This compound is soluble in water and is used directly in the dyeing operations.

Eulan NK



Eulan AL



This product is also water soluble and will withstand ten washings or cleanings. Dr. Bayer stated that it was the best of the Eulans.

This Eulan is gasoline soluble and was manufactured and sold in large quantities during the war.

Dr. Bayer was interviewed.

10. LIST OF DOCUMENTS

The following documents were sent to MIRS for filing:

- a. No. III I.G. Leverkusen
"Method of Preparation of a Number of Products"
- b. No. XIV I.G. Leverkusen
"Process for Making Lauseto and Directions for
Impregnation of Clothing."
- c. Folder No. 25, Dr. Schonhöfer - Elberfeld.

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